

THERMO-HYDROLOGIC LABORATORY STUDIES SAMPLE COLLECTION AND CHEMICAL ANALYSES

PROCEDURE ID: YMP-LBNL-TIP/TT 13.0 | Rev. 0, Mod. 1

EFFECTIVE: 8/25/00

1. PURPOSE

This Technical Implementing Procedure (TIP) provides instructions for the collection of liquid samples and the measurement of chemical constituents in water samples for Thermo-Hydrologic Laboratory Studies at Lawrence Berkeley National Laboratory (LBNL) for supporting the Yucca Mountain Site Characterization Project (YMP).

2. SCOPE

This procedure applies to all LBNL personnel (or contractor personnel following LBNL procedures) involved in work performed as described in Master Planning Document (MPD) YMP-LBNL-MPD-THL-1.0, Thermohydrologic Laboratory Studies -FY 99 or applicable subsequent planning document, when this work is not covered While this procedure incorporates specific under other YMP-LBNL-TIPs. requirements unique to the laboratory studies, it is consistent with the requirements described in YMP-LBNL-QIP-SII.0, Documenting Sample Control.

For all technical activities, data collected using this procedure and any equipment calibrations or recalibrations that may be required shall be in accordance with this TIP and in full compliance with YMP Administrative Procedure (YAP)-12.3Q, Control-All documentation of Measuring and Test Equipment and Calibration Standards. resulting from actions taken under this TIP will be recorded in Scientific Notebooks and Equipment Log Books as described in the Office of Civilian Radioactive Waste Management (OCRWM) Administrative Procedure (AP)-SIII.1Q, Scientific Notebooks. Electronic data maintenance, controls and transfers shall be controlled in accordance with YMP-LBNL-Quality Implementing Procedure (QIP)-SV.0, Control of the Electronic Management of Data.

If this procedure cannot be implemented as written, YMP-LBNL personnel shall notify the responsible Principal Investigator (PI) or designee. If it is determined that a portion of the work cannot be accomplished as described in this TIP, or would produce undesirable results, that portion of the work shall be stopped and not resumed until this procedure is modified per YMP-LBNL-QIP-5.2, Preparing Development Plans & Quality / Technical Implementing Procedures.

If the responsible PI or designee determines that a modification or a revision to the TIP would cause an unreasonable delay in proceeding with the task, then an expedited change to the procedure, including documentation of deviation from the approved procedure, can be made according to YMP-LBNL-QIP-5.2. Such changes

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are subject to review, usually after the task has proceeded, and thus work performed under TIPs with expedited changes is done at risk of future invalidation.

Employees may use a controlled electronic or hard copy of this procedure; however, employees are responsible for assuring that the correct revision of this procedure is used. When this procedure becomes obsolete or superseded, it must be destroyed or marked "superseded" to ensure that this document is not used to perform work.

3. PROCEDURE

3.1 Sample Collection

Samples for chemical analysis shall be collected in high-density polyethylene bottles deemed suitable for collection and storage of samples. Samples shall be refrigerated for later delivery to the Environmental Measurement Laboratory at LBNL.

3.1.1 Sample Name/Bottle Labeling

Samples shall be given a unique identifier such as "YMP-LBNL-THL-date-sample number" or an appropriate abbreviation thereof. Sample names shall be marked with an indelible laboratory marker either directly on the bottle or on an adhesive sticker affixed to the bottle along with the name of the originator and the date. This information shall be entered into the scientific notebook as well as the time of collection, location of sample collection, approximate size of the sample, any conditions which may affect the outcome of the sampling and analysis such as approximate sample temperature and amount of headspace. A hazard label shall be placed on the bottle identifying potential hazards.

3.1.2 Sample Collection

Samples shall be collected from appropriate locations in the experimental system such as the reservoir downstream of the tuff dissolution column (influent to the fracture) or other locations as needed. The frequency of sampling shall be determined by the PI based on data needs and system changes. Initial sample collection frequency shall be several times daily. Later in the experiment, sample collection frequency may be weekly.

Samples shall be siphoned from the reservoir and collected in an open (plunger removed) syringe with an attached 0.2 micron filter at the outlet with a minimum of disturbance allowing the flowing sample to

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run down the side of the syringe. Upon obtaining the required volume (see below) the plunger shall be inserted and the sample filtered and collected in sample bottles allowing the filtered liquid to flow down the side of the bottle with a minimum of disturbance until the bottle overflows. The bottle shall be capped immediately with a minimum of headspace. Since many samples will be collected hot, the person collecting the sample shall take appropriate precautions (i.e. wear appropriate gloves such as standard cotton or leather gloves or use some other method of handling the hot bottles).

Six water samples shall be collected as described above. One 60 ml sample will be used for anions. The second 60 ml sample shall be acidified with 200 microliters of concentrated nitric acid. Appropriate gloves (latex or nitrile) shall be worn during the acidification process. Two 30 ml samples will be used for alkalinity. The remaining samples shall be collected in 4 - 8 ml bottles, sealed, and immediately placed in a room temperature water bath. pH and electrical conductivity will be measured in these samples as discussed in Section 3.2.

The size of sample collected shall be at least 196 ml as described above unless additional or less sample is required for the analyses, in which case, appropriate sized samples shall be collected. The minimum sample size shall be the minimum size required for the analyses considering the sizes of the sample bottles available. If adequate fluid is present in the reservoir, a duplicate sample shall be collected and labeled as another sample. The duplicates will be tracked as the other samples, but will be analyzed as blind duplicates at the discretion of the PI to check laboratory performance. No indication that a sample is a duplicate will be made on the bottle, however, identifying information required in 3.1.1 will be kept in the scientific notebook.

3.1.3 Sample Bottles

High-density polyethylene sample bottles with tight sealing caps shall be used. Samples for electrical conductivity and pH shall be collected in clean glass or high-density polyethylene tubes.

3.1.4 Sample Handling/Preservation

Following sample collection, samples shall be immediately refrigerated for later delivery to the Environmental Measurement Laboratory. All samples shall be analyzed within one (1) month of collection, except alkalinity, which shall be measured within one day of sample collection. If samples cannot be analyzed within the

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required timeframe, they shall be analyzed at the first available opportunity, and a notation shall be placed in the scientific notebook identifying the duration samples have exceeded the analysis timeframe. A notation shall also be placed on the analysis results allowing special consideration to be given to data generated, and an analysis of the data applicability. This analysis shall be documented in the scientific notebook.

3.1.5 Sample Transfer

Samples shall be transferred from the laboratory where they were collected to the Environmental Measurement Laboratory at LBNL along with a chain-of-custody form (Attachment 5) containing the sample identification, time of collection, sample condition indicating the presence of headspace, and analyses required. The samples shall be transported in a plastic cooler so that the temperature of the samples is maintained for the short walk between laboratories. A copy of the chain-of-custody form shall be placed in a scientific notebook, and a copy shall be left with the samples. Signatures of the person delivering the samples and the person receiving the samples, and the time and date of transfer shall be placed on the chain-of-custody form. The laboratory may assign another tracking number to the samples if necessary, and this identification shall be placed on the chain-of-custody form next to the originally given name to provide traceability.

3.1.6 Chemical Analysis Checks

Samples shall be retained under refrigeration until all checks are completed to ensure the quality of the chemical analyses. Such checks may include the use of a distribution of species code such as EQ3/6, comparison of electrical conductivity vs. charged species, and pH vs. alkalinity.

3.1.7 Sample Disposal

Following analysis and quality checks (Section 3.1.6), samples no longer required shall be disposed of following accepted laboratory practice. Disposal shall be recorded in the laboratory notebook.

3.2 Analytical Techniques

Silica, calcium, sodium, potassium, aluminum, magnesium, iron, strontium, and manganese shall be measured using Environmental Protection Agency (EPA) Method 6010B (Revision 2, December 1996, See Attachment 1) as

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described in SW 846 (EPA). Acidified samples shall be used for these analyses. If adequate sensitivity is not obtained using this method for aluminum (nondetectable concentrations), EPA Method 6020 (Revision 0 September 1994, See Attachment 2) shall be used to determine aluminum concentration at the discretion of the PI. Analyses for boron will be made at the discretion of the PI using the ICP-MS method described in EPA Method 6020.

Anions (Cl⁺, F, SO₄⁻, and NO₃) shall be measured using ion chromatography as specified in Standard Methods 4110 (See Attachment 3, American Public Health Association et al.). If fluoride cannot be accurately measured using this technique (i.e. the fluoride peak cannot be adequately separated from the carrier peak), an alternate technique will be found. Non-acidified samples shall be used in these analyses.

Total alkalinity shall be measured using Standard Methods 2320 (See Attachment 4, American Public Health Association et al.). Non-acidified samples shall be used in this analysis.

pH and electrical conductivity shall be measured immediately after sampling when the sample is cooled to room temperature. Following collection, the samples shall be immediately sealed and placed in a water bath at room temperature until measurements are made. pH indications shall be made according to YMP-LBNL-TIP/TT 12.0, Thermo-Hydrologic Laboratory Studies Calibration, Measurement, & Electronic Data Handling Procedure and recorded in the scientific notebook every 30 seconds until a steady pH value is obtained. Electrical conductivity measurements shall be made by inserting the calibrated electrode into the sample tube, and reading the display. If the display is non-steady, values indicated by the instrument will be recorded in the scientific notebook every 30 seconds until a steady value is reached. The pH meter shall be calibrated (see Section 3.3.2.1) prior to use. The electrical conductivity meter shall be calibrated (see Section 3.3.2.2) prior to use. Non-acidified samples shall be used in these analyses.

3.3 Equipment Calibration

M&TE Justification forms shall be documented by the PI for each standard used to calibrate M&TE in accordance with this TIP per YAP-12.3Q. These forms shall be filed in the scientific notebook.

3.3.1 Identification of standards to be used

Calibration standards for pH, Electrical Conductivity, Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP), Inductively

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Coupled Plasma-Mass Spectrometry (ICPMS), and Ion Chromatography (IC) shall be traceable to the National Institute of Standards and Technology (NIST) in accordance with the QAP 2.0 Activity Evaluation contained in YMP-LBNL-TJK-4A pp. CH-1 to CH-22. Verification of NIST traceability shall be demonstrated by a certificate of analysis provided by the manufacturer or vendor showing the comparison of the standard purchased to the NIST standard. For ICP, ICPMS, and IC, NIST-traceable standards shall be procured from two manufacturers.

3.3.1.1 pH

Off-the-shelf buffers accurate to 0.01 pH units or better with NIST traceability are required. Certificates of Analysis are required showing the comparison between the lot and the primary NIST standard.

3.3.1.2 Electrical Conductivity

Off-the-shelf standards accurate to 0.3 % or better with respect to the stated concentration on the Certificate of Analysis, with NIST traceability are required. Certificates of Analysis are required showing the comparison between the lot and the primary NIST standard.

3.3.1.3 Inductively Coupled Plasma, Inductively Coupled Plasma-Mass Spectrometry

Off-the-shelf standards prepared from ultra-high purity grade chemicals or metals (99.99% or greater) shall be used for calibration. All standards shall be traceable to NIST. Additionally, a second NIST-traceable standard of similar quality obtained from a different manufacturer shall be used to verify calibration standards. Thus, two independent standards will be used, one in calibration of the equipment, and the other in verifying the calibration. Certificates of Analysis are required showing the comparison between the lot and the primary NIST standard.

3.3.1.4 Ion Chromatography

Off the shelf standards with NIST traceability are required. A second independent standard will also be used to verify the calibration of the IC equipment. As with the ICP methods, these standards will be obtained from a standard source different from that of the calibration standard and at concentrations within the

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linear working range of the instrument. Thus, two independent standards will be used, one in calibration of the equipment, and the other in verifying the calibration. Certificates of Analysis are required showing the comparison between the lot and the primary NIST standard.

3.3.2 Detailed Description of Calibration Method

3.3.2.1 pH

pH meter calibration shall be performed according to standard calibrating techniques in which the pH probe is set in a particular buffer and the meter indicator is changed to match the pH of the buffer. This process is repeated using buffer solutions of different pH calibrating the instrument such that the instrument reads the proper pH for the buffers used. Multiple standards shall be used bracketing the range of pH values expected. For one specific meter, an example of pH calibration is found in YMP-LBNL-TIP/TT 12.0, R1, Mod 0, Thermohydrologic Laboratory Studies, Calibration and Measurement. Measurements shall be made within two hours of calibration.

3.3.2.2 Electrical Conductivity

The cell constant shall be determined using a calibration standard. The conductivity meter shall be set such that the meter indication is equal to the conductivity of the standard. If this is not possible, a calibration curve may be generated. Such a curve is constructed by plotting instrument response against the certified values of standards. A curve is fit to the values obtained, and an equation is determined for the curve. The equation of the plotted calibration curve can then be used to calculate values of unknown solutions between the calibration points. All calibration values and curves shall be placed in the scientific notebook. Calibration shall be performed annually and checked prior to each measurement.

3.3.2.3 Inductively Coupled Plasma (ICP)

The detailed calibration method for ICP is contained in Sections 7 and 8 of Attachments 1 and 2.

3.3.2.4 Ion Chromatography (IC)

3.3.2.4.1. Prior to calibration and use, IC equipment shall be turned on and warmed up for at least 30 minutes.

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Calibration shall be accomplished by conducting 3.3.2.4.2. measurements on a series of standard solutions that span the expected range of concentrations of the unknowns. These measurements shall be made in exactly the same manner as samples. Mixtures of standards are suitable for calibration. The method of preparation of the mixture shall be recorded in the scientific notebook with the masses or volumes of all components identified. A response (e.g. peak area or height) shall be obtained for each standard, and then these responses and the known concentrations shall be used to construct a calibration curve (linear or quadratic). A minimum of four concentrations for standard solutions and a calibration blank is required to establish the calibration curve. The curve is then used to determine the concentrations of unknowns given their responses. In order for the calibration curve to be acceptable, the correlation coefficient (R2) must be equal to or greater than 0.998. If the data does not meet this criterion, actions shall be taken to correct the problems [e.g., rerun or remake the standard(s), check/modify the experimental conditions, check the instrument].

Calibrations shall be performed at the beginning of each set of sample measurements and verified at the end of the set (or at interval that depends on the experience that the PI and/or TIP users have with the instrument). The results of all calibrations and the times at which they were conducted shall be recorded in scientific notebooks in accordance with AP-SIII.1Q and YAP-12.3Q.

- 3.3.2.4.3. Calibration verification shall be performed using a NIST-traceable independent standard obtained from a source other than the source of the calibration standard. The calibration verification standard shall be introduced within the linear range of the IC equipment, and run following the initial calibration of the equipment.
- 3.3.2.4.4. Documentation shall be included in the scientific notebook as stated in Section 3.3.6.
- 3.3.3 Consideration of Manufacturer's Recommendations for Storage and Handling

M&TE shall not be handled in a manner which adversely affects its current or future performance. All M&TE covered by this procedure

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shall be used in laboratory environments, and stored at room temperature. pH probes shall not be used to vigorously stir solutions. The electrical conductivity probe shall be rinsed off with distilled water before and after use and allowed to dry in air. Solutions causing fouling of IC and ICP equipment shall not be used in these M&TE.

3.3.4 Identification of Tolerances and Ranges of Use

Calibration curves shall be used to define the tolerances and range of use for M&TE covered by this TIP. For pH measurement, the tolerance shall be the tolerance of the buffer solutions used. For electrical conductivity, the tolerance shall be the tolerance of the standard used for setting the cell constant or the standards used to determine the calibration curve. For IC and ICP, the R² value of the calibration curve shall define the tolerance.

3.3.5 Identification of Calibration Intervals

3.3.5.1 pH

Calibration shall be performed each time samples are analyzed.

3.3.5.2 Electrical Conductivity

Calibration shall be performed annually, with operational checks performed each time samples are analyzed.

3.3.5.3 ICP

Calibration shall be performed each time samples are analyzed.

3.3.5.4 IC

Calibration shall be performed each time samples are analyzed.

3.3.6 Documentation

Staff Members shall document the following information in the scientific notebook or on the M&TE Calibration Documentation Form (Attachment 6) with the calibration results:

- a) The unique identification of the M&TE calibrated
- b) Date and time calibrated

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- c) Calibration data, and results of the calibration and statement of acceptability
- d) Re-calibration due date or calibration interval/frequency
- e) Procedure (including revision level) used to calibrate the M&TE
- f) Identification of and traceability to the calibration standards used for the calibration
- g) As-found condition of the M&TE, as appropriate
- h) Specified range and tolerances and whether the M&TE met those tolerances
- i) Personnel performing calibrations
- j) Reference to actions taken with out-of-calibration or non conforming M&TE, including evaluation results, as appropriate.

3.3.6.1 Calibration Stickers

Calibration stickers shall not be affixed to ICP, IC, and pH equipment, as calibration is required each time samples are analyzed, and calibration is an integral part of the measurement procedure. A calibration sticker shall be affixed to the electrical conductivity meter stating the unique identifier of the meter (manufacturer, model, serial number), date of the calibration, next calibration date and calibrator's name, and calibration standard used.

3.3.7 Controls for Nonconforming or Out-of-Tolerance Conditions

Nonconforming or out-of-tolerance equipment shall be segregated or tagged with an Out of Service tag and not be used. Recalibration shall be attempted to remedy the nonconformance. If this is ineffective, the nonconforming equipment shall be replaced if possible. If replacement is impossible or the replacement timeframe is such that sample degradation will occur rendering samples useless, nonconforming equipment may be used with notification of the PI and an evaluation of the effects of using such equipment on the data is performed and documented on the M&TE Out of Calibration Report (OCR) as described in YAP-12.3Q. The OCR shall be filed in the scientific notebook. If it is determined that the data is impacted, a Nonconformance Report (NCR) shall be initiated in accordance with

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YAP-15.1Q.

3.3.8 Recalibration When Updates to Software Contained Affects Calibration

All equipment relying on software shall be recalibrated following installation of software updates which affect the calibration. All software used in M&TE is integral to the M&TE.

3.3.9 Staff Member shall document each usage of the equipment on the M&TE Standard Usage Log as described in YAP-12.3Q or the same information, at minimum in the scientific notebook.

4. RECORDS

4.1 Lifetime

Records generated as a result of this TIP are entries in:

- · Scientific notebooks or attachments to such notebooks,
- Equipment Logbooks (including MT&E Justification Forms and Standard Usage Log)
- MT&E Out of Calibration Reports, if applicable.

4.2 Non-Permanent

None

4.3 Controlled Documents

This Technical Implementing Procedure

4.4 Records Center Documents

Records associated with this procedure shall be submitted to the Records Processing Center in accordance with AP-17.1Q, Record Source Responsibility for Inclusionary Records.

5. RESPONSIBILITIES

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- 5.1 The Principal Investigator (PI) is responsible for assuring full compliance with this procedure and providing training thereof. The PI is responsible for overseeing and coordinating the preparation, review, distribution, revision, and recommending rescission of the TIP.
- 5.2 Staff Members involved in thermal-hydrologic laboratory studies are responsible for following this procedure and turning over related documentation to the Records Coordinator for submittal to the Records Processing Center in accordance with AP-17.1Q. Related data shall be turned over to Technical Data Coordinator for submittal to the YMP Technical Data Management System (TDMS) in accordance with AP-SIII.3Q, Submittal and Incorporation of Data to the Technical Data Management System.

6. ACRONYMS AND DEFINITIONS

6.1 Acronyms

AP OCRWM Administrative Procedure

EA Engineering Assurance

EPA Environmental Protection Agency

IC Ion Chromatography

ICP Inductively Coupled Plasma

ICPMS Inductively Coupled Plasma Mass Spectrometry

LBNL Lawrence Berkeley National Laboratory

M&TE Measuring and Test Equipment

NCR Nonconformance Report

NIST National Institute of Standards and Technology

OCR Out of Calibration Report

OCRWM Office of Civilian Radioactive Waste Management

OQA Office of Quality Assurance

PI Principal Investigator

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QIP Quality Implementing Procedure

TIP Technical Implementing Procedure

TDB Technical Data Base

YAP YMP Administrative Procedure

YMP Yucca Mountain Site Characterization Project

6.2 Definitions

Staff Member: Any scientist, engineer, research or technical associate, technician, or student research assistant performing quality-affecting work for YMP-LBNL.

Technical Implementing Procedure: Each TIP describes YMP-LBNL technical and/or scientific tasks that (1) are repetitive, (2) are standardized, and (3) can return different results if deviation from the sequence of steps occur.

7. REFERENCES

AP-17.1Q, Record Source Responsibility for Inclusionary Records

AP-SIII.1Q, Scientific Notebook

AP-SIII.3Q, Submittal and Incorporation of Data to the Technical Data Management System

YAP-12.3Q, Control of Measuring and Test Equipment and Calibration Standards

YAP-15.1Q, Control of Nonconformances

YMP-LBNL-MPD-THL-1.0, Thermo-Hydrologic Laboratory Studies - FY99

YMP-LBNL-QIP-5.2, Preparing Development Plans & Quality/Technical Implementing Procedures

YMP-LBNL-QIP-SII.0, Documenting Sample Control

YMP-I BNI -QIP-SV.0, Control of the Electronic Management of Data

YMP-LBNL-TIP/TT 12.0, Thermo-Hydrologic Laboratory Studies Calibration, Measurement, & Electronic Data Handling Procedure

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U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3 ed., Final Update III

American Public Health Association, American Water Works Association, Water Environment Federation, "Standard Methods for the Examination of Water and Wastewater, 18th ed., 1992.

8. ATTACHMENTS

Attachment 1:

Method 6010B, Inductively Coupled Plasma-Atomic

Emission Spectroscopy, EPA SW 846.

Attachment 2:

Method 6020, Inductively Coupled Plasma-Mass

Spectrometry, EPA SW 846.

Attachment 3:

4110. Determination of Anions by Ion Chromatography,

American Public Health Association et al..

Attachment 4:

2320. Total Alkalinity American Public Health Association et

al.

Attachment 5:

Chain of Custody Record

Attachment 6:

Measuring and Test Equipment (M&TE) Calibration

Documentation Form

9. REVISION HISTORY

12/03/99,

Revision 0, Modification 0

Initial issue.

8/25/00,

Revision 0, Modification 1

Minor editorial changes

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10. APPROVAL Preparer: T. J. Kneafsey	8/24/2000 Date:
F.A Reviewer: N. Aden-Gleason	8/23/2668 Date:
OQA Concurrence: S. Harris	<u>8/23/00</u> Date:
Principal Investigator: K. Pruess	8/23/00 Date:
Project Manager: G. S. Bodyarsson	Date:

YMP-LBNL-TIP/TT 13.0, Rev. 0, Mod. 1 Attachment 1

Inductively Coupled Plasma-Atomic Emission Spectrometry, EPA SW 846

This document is not available electronically. A copy is available in the EA office, in 90P.

YMP-LBNL-TIP/TT 13.0, Rev. 0, Mod. 1 Attachment 2

Method 6020, Inductively Coupled Plasma-Mass Spectrometry, EPA SW 846

This document is not available electronically. A copy is available in the EA office, in 90P.

OFFICE OF	CIVILIAN RADIOACTIVE WASTE MANAGEMENT				
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SPECIAL HANDLING INSTRUCTIONS					
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Document Requiring Special Handling	Att. 3: Method 4110. Determination of Anions by Ion Chromatography. Copyright 1992 by the American Public Health Assoc.				
Document Revision/Chan	ge:RO MO				
Document Date:	12/03/1999				
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Document Requiring Special Handling	Att. 4: Method 2320. Total Alkalinity. Copyright 1992 by the American Public Health Assoc.				
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Document Date:	12/03/1999				
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Chain of Custody Record

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Measuring and Test Equipment (M&TE) Calibration Documentation Form

a) M&TE Description	b) M&TE Unique Identification	c) Calibration Date and Time (if applicable)
d) Person Performing Calibrations		e) M&TE Condition (As-Found) Working
		Not Working
f) Calibration Procedur level)	e (including revision	g) Calibration Standards Used
h) Location of Calibrati	on Data	i) Location of Calibration Results
YMP-LBNLPage(s)		YMP-LBNLPage(s)
k) Specified Range and	Tolerances	
l) Re-calibration due da interval/frequency	te or calibration	m) Reference to actions taken with out-of-calibration or non conforming M&TE, including evaluation results, as appropriate YMP-LBNL Page(s)
n) Comments		1 460(3)
Signature		Date